**FINAL REPORT—BERKELEY PIT
INNOVATIVE TECHNOLOGIES
PROJECT, PHASE II: METRE
GENERAL INC., DEMONSTRATION**

**MINE WASTE TECHNOLOGY PROGRAM
ACTIVITY IV, PROJECT 7**

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Federal Energy Technology Center
Pittsburgh, Pennsylvania 15236
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Foreword

Today, industries are developing and modifying technologies to produce their products more efficiently. The waste generated by these industries, if improperly dealt with, can threaten public health and degrade the environment. The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the EPA strives to formulate and implement actions leading to a balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define, measure the impacts, and search for solutions to environmental problems.

EPA's National Risk Management Research Laboratory (NRMRL) is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis. This supports the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. The Federal Energy Technology Center (FETC) of the U.S. Department of Energy (DOE) has responsibilities similar to the NRMRL in that FETC is one of several DOE centers responsible for planning, implementing, and managing research and development programs. In June 1991, an Interagency Agreement (IAG) was signed between EPA and DOE that made funds available to support the Western Environmental Technology Office's operating contractor, MSE Technology Applications, Inc. (MSE), and Montana Tech of The University of Montana for the development of the Mine Waste Technology Program (MWTP). This publication is one of the products of the research conducted by the MWTP through these two Federal organizations and provides a vital communication link between the researcher and the user community.

The objective of this demonstration was to remediate Berkeley Pit water to the extent that it could be safely used for agricultural applications, to demonstrate the suitability of the technology in allowing continuous online treatment that could be scaled up to match present inflows into the Berkeley Pit, and to evaluate the possible recovery of the economic minerals in the compacted precipitate.

Executive Summary

The Berkeley Pit Innovative Technologies Project (BPIT) tests innovative technologies that can be used to treat the acidic, heavy metal contaminated flows collecting in the Berkeley Pit. Metre General Inc. (MGI) tested the use of a combination of conventional and innovative technologies as a potential solution to the problem of treating the Berkeley Pit water.

The innovative technology is the commercial product Octolig™, which can be used to remove heavy metals from aqueous media. Octolig™ is manufactured by immobilizing ligands (amines and imines) on a silica gel substrate using a proprietary process. The immobilized ligands have an extremely high affinity for heavy metals.

MGI tested the combination of the Octolig™ technology with a conventional precipitation process.

The specific goals for the proposed process were:

  C to remove copper from the wastewater as a recyclable metal using Octolig™;
  C remove the bulk of the metals as hydroxides using an alkaline precipitation process; and
  C achieve the low effluent target concentrations for as many of the parameters as possible with a polishing step using Octolig™.

Each of these three goals was tested with a separate process step. MGI's investigators performed the testing in the laboratory facilities at the Montana Tech of the University of Montana from October 20th to 24th, 1997. All sampling and analyses were performed according to the Project Quality Assurance Plan.

The project met most of the water quality goals. The water quality targets for aluminum and manganese were not met in two of the three experiments. The goal of removing copper as a recyclable metal was only partially successful. Although all of the copper was recovered, some of the iron, aluminum, zinc, and manganese were also recovered with the copper. The recovered product will have a copper concentration of 30% or more.

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Tables

1. Introduction

The Berkeley Pit Innovative Technologies (BPIT) Project tests innovative technologies that can be used to treat the acidic, heavy metal- contaminated flows collecting in the Berkeley Pit. Metre General Inc. (MGI) was selected to test their proposed process in the laboratory facilities of Montana Tech of the University of Montana (Montana Tech).

MGI proposed the use of a combination of conventional and innovative technologies for treating the Berkeley Pit water. The innovative technology is the commercial product Octolig™ which can be used to remove heavy metals from aqueous media. Octolig™ is capable of removing metals down to extremely low concentrations in the order of 10 ppb.

Octolig™ is manufactured by immobilizing ligands (amines and imines) on silica gel particles using a proprietary process (Eaglen). The immobilized ligands have an extremely high affinity for complexation with heavy metals. Metal ions form complexes with the immobilized ligands and are thus removed from solution. MGI proposed a process that combines the Octolig™ technology with a conventional precipitation process in response to the BPIT Request for Proposal (RFP).

2. Project Goals and Objectives

The specific goals for the proposed process configuration as well as the testing protocol were:

- C To recover copper from the Berkeley Pit water as a recyclable metal using Octolig™.

- C To remove the bulk of the metals as hydroxides using a precipitation process.

- C To achieve the low effluent target concentrations for as many of the parameters as possible using a final polishing treatment step using Octolig™.

MGI did not propose a technology for removal of sulfate because the sulfate can be removed by conventional precipitation techniques that will produce a nonhazardous gypsum sludge after all the metallic contaminants have been removed.

3. Process Technology

Based on the data presented in the RFP, the Berkeley Pit water was observed to contain high concentrations of metals such as aluminum, copper, iron, manganese, and zinc with values of several hundred parts per million and significant concentrations of cadmium, nickel, and arsenic (Table 3-1) and a pH of 2.85. The high concentrations of these contaminants of concern coupled with the low target values for effluent concentration present a treatment problem.

The removal of bulk metals and sulfates is most economically performed using conventional technologies like neutralization and precipitation. These technologies yield large quantities of sludge as a waste byproduct. However, precipitation technologies do not achieve the low target concentrations (Table 3-1) for all the contaminants of concern.

The proposed process scheme is based on the strategy of optimizing the advantages of conventional precipitation technologies as well as the Octolig™ process to attain the treatment goals for the Berkeley Pit water. A three step process was tested involving: 1) recovery of copper from the Berkeley Pit water using Octolig™; 2) removal of bulk concentrations of all metals using conventional lime precipitation; and 3) treatment of the water with Octolig™ to achieve the target effluent concentrations in Table 3-1.

3.1 Octolig™ Technology

The Octolig™ technology is being proposed to achieve two objectives: 1) remove the majority of the dissolved copper as a recyclable stream; and 2) achieve the low target concentrations for the heavy metals after the majority of the metals have been removed as metals hydroxides using conventional precipitation technology.

The commercial product Octolig™ (U.S. Patent No. 5,190,660) is manufactured by immobilizing branched polyethylene imine (average molecular weight 12,000) on silica gel. The silica gel is a commercial product, which is sieve-sized so that 95% of product passes through 35 mesh and is retained by 60 mesh. The product has the physical characteristics of silica gel and retains all of the chemical activity of branched polyethylene imines (BPEI).

Table 3-1. Table of Elements, Concentrations, and Target Concentrations in the Berkeley Pit Water

Contaminant	Current Concentration (mg/L)	Target Concentration (mg/L)
Aluminum	260	0.05
Arsenic	0.8	0.05
Cadmium	2.14	0.03
Chromium	0.078	0.05
Copper	172	0.01
Iron	1068	0.3
Manganese	185	0.05
Nickel	0.9	0.1
Zinc	550	0.1

| pH* | 2.85 | 6.5 to 8.5 |

* pH is in standard pH units

Branched polyethylene imines form stable complexes with many metal ions. The polyimine structure contains repeating amine groups that have a high affinity for heavy metal ions. Extensive research performed on the metal loading behavior of BPEIs immobilized on silica gel demonstrates that loading is proportional to the nitrogen group density on the surface of the silica gel. For example, for Cu^{2+}, it was observed that the ratio of maximum copper loading to nitrogen groups was 1:2, two nitrogen groups complexing with one copper. Polyethylene imines have the ability to form complexes with a wide variety of metal ions including Cu^{2+}, Zn^{2+}, Ni^{2+}, Mn^{2+}, Co^{2+}, Cd^{2+}, and Pb^{2+}.

The complex formation is also pH dependent for most metals; in strongly acidic solutions, as the amine groups are protonated very little complexation occurs; and in alkaline solutions there is a competition between hydroxy groups OH^- and amines for complexation with metal ions. The exception to this rule is copper, and to some extent, arsenate, both of which are complexed at low pHs. The intermediate pH range of 4.0 to 9.0 is the most appropriate for the formation of polyethylene imine-metal complexes.

Octolig™ has been successfully applied in the past for removal of metals such as copper, nickel, and cadmium from electroplating wastewater. The product has been tested for a multitude of applications (Ref. 1) as part of a comprehensive research program.

3.2 Precipitation Technology

Precipitation technology is based primarily on the pH relation of solubility of metal ions. Metal ions are soluble in acidic water and have very

low solubilities at neutral and alkaline pHs. As pH of acidic water is increased by addition of an alkaline reagent, metal ions precipitate as metal hydroxides.

The process consists of five steps: 1) addition of neutralizing reagent (e.g., lime); 2) aeration of solution to oxidize all metals; 3) addition of flocculating agent; 4) clarification of water by settling out precipitated sludge in a clarifier; and 5) removal of metals as a slurry from the underflow of the clarifier and thickening or dewatering the sludge.

The major advantage of conventional precipitation technologies is that it is a proven, inexpensive method for the consistent removal of high concentrations of metals. The disadvantages are that the process generates high volumes of sludge, which may require disposal at a regulated repository; the process can also be limited in achieving low concentrations of metals (less than 1 ppm) by the solubility equilibria of metals as well as the engineering limitations of physically separating precipitated sludge from the water.

The disadvantages of having a high volume of sludge have been overcome to some extent in industrial applications by recycling the sludge. This increases the density of the underflow from the clarifier, or the sludge thickener. This process has been successfully applied by all the major water treatment plant manufacturers. The theoretical basis for the increase in sludge density with recycle is explained according to the Kynch theory. This process has been successfully applied at the Summitville Mine (a Superfund mine cleanup in Colorado) for treatment of acidic mine drainage very similar in composition to the Berkeley Pit waters.

4. Process Configuration

The process that was tested has three stages. Each stage has a definite goal and a purpose in the overall process scheme. The following is a brief description of each stage.

4.1 Stage I: Removal of Copper

The Berkeley Pit water is first treated through a column of Octolig™ at the pH of the raw water. The column, filled with a packed bed of Octolig™, will be operated in the down-flow mode. At this pH, Octolig™ has very little affinity for any metal other than copper. It is expected that the Octolig™ will remove as much as 80% to 90% of the copper present in the water in one pass. This column of Octolig™ containing copper complexed with ligands is regenerated with two bed volumes of 4% nitric acid solution to produce an effluent with a high concentration of copper. This copper can be reclaimed using a variety of technologies including electrowinning.

4.2 Stage II: Neutralization and Precipitation

The second stage of the process is designed to remove the majority of the metals including the remaining copper, iron, aluminum, zinc, and manganese. This process stage is configured according to other successful conventional treatment processes with the following steps:

- C Addition of hydrated lime to the water to increase the pH to 8.5.

- C Simultaneous aeration of the water to oxidize the iron present as ferrous to the ferric state.

- C Addition of a small dosage of anionic polymer to improve flocculation and settling properties of metal hydroxides that precipitate from the solution as the pH is increased.

- C Clarification of the water to obtain an effluent free of suspended solids.

The objective of this process stage is to remove as much of the bulk metals as possible and obtain a treated water that contains concentrations of metals ranging from a few parts per million for manganese and zinc, to less than one part per million for iron, aluminum, copper, nickel, arsenic, and cadmium.

4.3 Stage III: Polishing with Octolig™

The third stage of the process will again utilize Octolig™ technology as a polishing step to remove all the metals to their target effluent concentrations as specified in Table 3-1. The treated Berkeley Pit water from Stage II will be treated through a second Octolig™ column, once again operated in the down-flow mode. It is expected that all of the metals will be removed in one pass through the column.

The target concentrations for the proposed configuration will be the concentrations listed in Table 3-1 or lower for all parameters except sulfate. Octolig™ is not expected to have an impact on sulfate concentrations. As suggested earlier, other technologies may be considered, which will remove the sulfate as a byproduct (such as gypsum).

5. Bench-Scale Testing Protocol

The bench-scale testing essentially mimicked the full-scale treatment process. The Stage I and Stage III processes for the Octolig™ were carried out as a continuous process using scaled down columns that simulate the full-scale process. The simulation of the neutralization and precipitation process was carried out as a batch process.

Octolig™ was loaded into two identical columns that were 4 inches in diameter and 32 inches in total length. The bed depth of the Octolig™ in both columns was 19 inches (1.78 kg of Octolig™ was used in each column). One column was set up for Stage 1 of the process and the second columns was set up for Stage III. Peristaltic pumps capable of pumping flow rates in the range of 0 to 1 gallon per minute per square foot (gpm/ft^2) were provided by MGI along with the test columns. These pumps were connected to the test columns using Tygon™ tubing.

5.1   Stage I

To prepare the columns for Stage I, the Octolig™ was rinsed with deionized water at a pH adjusted to 3.0. This was done to ensure that the Octolig™ was at the same pH as the Berkeley Pit water. This prevents any buffering reaction between the Octolig™ and the treated water. The column rinsate was at a pH of 3.3 for the first test and at a pH of 2.6 for the second and the third test.

Five gallons of Berkeley Pit water were then pumped through the first column at a flow rate of 0.5 gpm/ft^2 [166 milliliter per minute (mL/min)]. The effluent from the columns was collected in a 5-gallon jug. Three spot samples were collected from the effluent of the column for analysis of all the contaminants of concern.

At the end of the test, the Octolig™ column was regenerated by washing with 8.0 liters of 4% (by weight) nitric acid solution at a flow rate of 0.5 gpm/ft^2. The regeneration solution from the column was analyzed for all metals to demonstrate a mass balance over the entire test.

5.2 Stage II

The remaining effluent was pH adjusted to 8.5 to 8.8 while being aerated and agitated with a magnetic mixer. At a pH of 8.5, a small dosage [equivalent to 1 to 2 milligrams per liter (mg/L) of anionic polymer was added to enhance flocculation. The precipitated particles were allowed to settle out and the 5-gallon clear solution was decanted. The solids slurry was filtered through a vacuum filter with a 41–micron filter. A sample of the clear water was analyzed for total metals (from Table 3-1).

5.3 Stage III

The clear solution (with a 5-gallon volume) from Stage II (at a pH between 8.5 and 9.0) was then pumped through the second column of Octolig™ at a flow rate equivalent to 0.5 gpm/ft^2 (166 mL/min). The effluent was again collected in a 5–gallon jug. Samples of the effluent were again collected and submitted for analysis of all the parameters in Table 3-1.

The entire test (all three stages) was repeated twice for a total of three tests in order to ensure reproducibility.

Sampling and analysis of samples was conducted according to a project quality assurance plan. All sampling, sample handling, shipping and analytical work was performed by the Project Manager for the BPIT Project.

6. Results and Discussion

The variables were kept constant over the three tests that were performed. The only variance observed, occurred in Stage I during the first test. The pH of the column started at 3.3 and migrated up to 8.3 by the end of the test. Brown precipitated particles could be observed forming in the column during the test. The Octolig™ product typically changes color from white to dark blue with copper complexation. This color change was restricted to the first 9 inches of the bed in the first test. This indicated that the initial pH of the column had to be reduced further in order to prevent the increase of pH of the treated water in the first column. Maintaining the low pH was critical because the intention of the bench- scale testing was to determine the amount of copper removed at the lower pH, without any neutralization.

In the second and third tests, the initial pH of the column was reduced to 2.6 through rinsing. The pH of the effluent from the column migrated from 2.6 to 2.9 during the course of the test. No precipitates could be observed and the color change to blue was observed to take place over the entire 19 inches of bed depth of Octolig™. These experiments were more representative of the first step of the process that had been proposed by MGI.

The tabulated data from the analysis performed at ACZ Laboratories is attached in the Appendix.

Table 6-1 is the analysis of the raw water samples from the Berkeley Pit prior to any treatment. A comparison with Table 3-1 shows that the concentrations of aluminum, copper, manganese, nickel, and zinc were significantly higher during the testing. The concentrations of iron and nickel are lower. Arsenic and chromium were not detected in the test samples, although they anomalously appear in later samples for Experiment #3 (Tables 6-3 and 6-4).

Table 6-2 presents the data for the effluent from the first Octolig™ column. This column was designed to remove the copper selectively at a low pH. The data for Experiment #1 shows that there was a significant pH variation in the first column. This anomaly was also observed and recorded during the experiment. The change in pH resulted in the total removal of almost all the metals except copper and zinc. In Experiments #2 and #3 the pH remained low. At the lower pH the Octolig™ removes significant fractions of aluminum, iron, manganese, and zinc, and reduces copper below its detection level. In Experiment #2, the concentratin of zinc is 0.3 mg/L, which is higher than the target concentration of 0.1 mg/L.

Table 6-3 represents the water quality of the effluent from Stage 1 treatment after it has undergone the neutralization, precipitation treatment in Stage 2. This is in effect the water quality that is being treated in the final Octolig™ column in Stage 3. This data shows that as proposed the precipitation step succeeds in removing almost all of the high concentrations of metals except for manganese in Experiment #2. This is to be expected at a pH of 8.1 since manganese is sometimes soluble up to a pH of 9.0 and higher. The pH in Experiment #3 appears to be an anomalous value, perhaps due to a sampling error (solid lime particles included in the sample). All the laboratory measurements showed a pH of 8.5 to 8.8 for this experiment.

Table 6-4 shows the final water quality data from all three experiments. There is a small rise in the chromium concentration for Experiment #3 (chromium was not detected in any prior samples). The best water quality was observed in Experiment #1, in which all the contaminants were reduced to below detection levels except for zinc. Even zinc was barely detected at its

Table 6-1. Analysis of Raw Berkeley Pit Water

	Sample Location: Berkeley Pit Water Sample		
Analyte	**Experiment #1**	**Experiment #2**	**Experiment #3**

pH (pH units)	2.5	2.5	2.6
Aluminum (mg/L)	341	299	290
Arsenic (mg/L)	<0.4	<0.4	<0.4
Cadmium (mg/L)	2.6	2.42	2.3
Chromium (mg/L)	<0.5	<0.2	<0.2
Copper (mg/L)	208	197	195
Iron (mg/L)	908	857	841
Manganese (mg/L)	248	230	225
Nickel (mg/L)	1.3	1.4	1.3
Zinc (mg/L)	703	659	642

Table 6-2. Analysis of Berkeley Pit Water After Stage 1 Treatment

Analyte	Sample Location: Effluent Sample Octolig™ Column		
	Experiment #1	**Experiment #2**	**Experiment #3**
pH (pH units)	8.3	2.8	3.3
Aluminum (mg/L)	<0.3	25.5	160
Arsenic (mg/L)	<0.4	<0.04	0.05
Cadmium (mg/L)	<0.03	0.64	1.89
Chromium (mg/L)	<0.1	<0.05	<0.1
Copper (mg/L)	0.2	<0.05	<0.2
Iron (mg/L)	<0.1	159	416
Manganese (mg/L)	<0.05	74.3	189
Nickel (mg/L)	<0.1	0.29	3
Zinc (mg/L)	0.1	189	545

Table 6-3. Analysis of Berkeley Pit Water After Stage 2 Treatment

Analyte	Sample Location: Sample After Precipitation Step		
	Experiment #1	Experiment #2	Experiment #3
pH (pH units)	7.9	8.1	11.7
Aluminum (mg/L)	0.6	0.6	0.2
Arsenic (mg/L)	<0.4	<0.04	<0.04
Cadmium (mg/L)	0.06	0.08	0.11
Chromium (mg/L)	<0.1	<0.05	0.1
Copper (mg/L)	0.1	0.11	0.2
Iron (mg/L)	<0.1	0.24	0.3
Manganese (mg/L)	116	26.7	0.1
Nickel (mg/L)	<0.1	<0.1	<0.1
Zinc (mg/L)	2.4	0.42	1.4

Table 6-4. Analysis of Berkeley Pit Water After Stage 3 Treatment

Analyte	Sample Location: Sample After Octolig™ Column		
	Experiment #1	Experiment #2	Experiment #3
pH (pH units)	3.3	8.6	8
Aluminum (mg/L)	<0.3	0.3	0.06
Arsenic (mg/L)	<0.4	<0.4	<0.04
Cadmium (mg/L)	<0.03	<0.03	<0.03
Chromium (mg/L)	<0.1	<0.1	0.1
Copper (mg/L)	<0.1	<0.1	<0.1
Iron (mg/L)	<0.1	<0.1	<0.1
Manganese (mg/L)	<0.05	0.59	4.29
Nickel (mg/L)	<0.1	<0.1	<0.1
Zinc (mg/L)	0.1	0.3	<0.1

minimum detection limit (MDL), and meets the target concentrations.

Table 6-5 contains the data for the regenerant solution from the regeneration of the Octolig™ in Stage 1 after each batch of treatment. As mentioned before, the regenerant solution consists of 8 liters of 4% nitric acid solution.

In Table 6-6 these concentrations have been used to perform a mass balance for all three experiments. The recoveries for metals is best in Experiment #3. On the average the best mass balance is observed for copper at 75%. The fluctuating and low recovery rates require further study. It is possible that the regeneration time used was not adequate enough to remove the metals.

Table 6-5. Analysis of Regeneration Solution of Octolig™ Column #1 (Stage 1 Treatment)

Analyte	Sample Location: Sample of Regenerant Solution of Octolig™ Column #1		
	Experiment #1	**Experiment #2**	**Experiment #3**
Aluminum (mg/L)	477	179	130
Arsenic (mg/L)	<2	<4	<4
Cadmium (mg/L)	3.8	1.1	1.1
Chromium (mg/L)	<0.5	<1	1
Copper (mg/L)	275	426	345
Iron (mg/L)	844	1030	846
Manganese (mg/L)	88.3	119	90.7
Nickel (mg/L)	2	1	1
Zinc (mg/L)	1020	346	260

Table 6-6. Mass Balance for Stage 1: Metals Recovery

Analyte	Experiment #1			Experiment #2			Experiment #3		
	grams/ removed	grams/ recovered	% recovery	grams/ removed	grams/ recovered	% recovery	grams/ removed	grams/ recovered	% recovery
Al	6.39	3.82	59.8	5.13	1.43	27.9	2.44	1.04	42.6
Cu	3.9	2.2	56.4	3.69	3.41	92.4	3.65	2.76	75.6
Mn	4.65	0.71	15.3	2.92	0.952	32.6	0.68	0.73	107.4
Fe	17.02	6.75	39.7	13.09	8.24	63	7.97	6.77	84.9
Zn	13.18	8.16	61.9	8.81	2.77	31.4	1.82	2.08	114.3

grams removed = (initial concentration - concentration after Stage 1)*3.75*5/1000
grams recovered = concentration in regeneration solution*8.0/1000

7. Conclusions

The experiments conducted at Montana Tech were successful in meeting the water quality goals of the project. Octolig™ achieved all the target concentrations in Experiment #1 and in Experiment #2, except for aluminum and manganese, and in Experiment #3 except for aluminum, manganese, and zinc. In Experiment #1, the pH in Stage 1 treatment was higher than in Experiments #2 and 3. The higher pH in Stage 1 is detrimental to the selective removal of copper but helps to achieve the water quality goals. In Experiment #1, there were essentially two high pH treatment steps through Octolig™ instead of one.

The attempt to recover copper without interference from other metals was not completely successful. Because the concentrations of zinc, iron, and manganese were too high relative to copper and reaction kinetics dictated that some of these metals also complex at lower pHs.

The potential for Octolig™ to be used as a polishing step for the treatment of Berkeley Pit water to achieve the water quality goals was demonstrated. The potential for recovering selective metals at a lower pH for recycling was also investigated and was only partly successful. The testing performed was too limited for the level of complications involved with the Berkeley Pit water to draw any definite conclusions.

The Octolig™ columns represent a low operating cost alternative for the reduction of metals concentration to extremely low concentrations consistently over a long period of time. The life of the Octolig™ product has been established to last over several hundred regeneration cycles.

Table 7-1. Projected Cost of Full-Scale Operation

Projected Cost of Full-Scale Operation 3 Million Gallons Per Day (MGD)[†]

Costs	Lime (88.110 #/day) @ $0.10/lb = $8.811/day

Yearly Costs

Lime	$3,216,000/yr
Labor	$400,000/yr
Maintenance	$1,000,000/yr
TOTAL	$4,616,000/yr
Octolig™ Material Replacement Cost	$6,300,000

[†] 3 MGD requires 90 each, 42 in dia. columns

8. References

1. Borup, Brett, et al., *Heavy Metal Removal from Domestic Wastewater Using Bound Macrocycles*, Department of Civil Engineering, Brigham Young University, Provo, Utah, Water Environmental Federation 1992.